



Burn-Rate Investigations of HAN-Based Candidate Liquid Propellants

by W. F. McBratney and J. A. Vanderhoff

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Abstract

High-pressure windowed strand burners have been used to obtain burning rates for some hydroxyl ammonium nitrate (HAN) liquid propellants over the pressure range from about 10 to 300 MPa. Measured linear burn rates for these liquids were erratic due to the manifestation of surface irregularities. This burning surface variable has been minimized by gelling. A pressure break occurs around 80 MPa for these gelled propellants. The burn rate (r) vs. pressure (p) can be adequately expressed by an exponential of the form $r = Ap^n$. For pressures below 70 MPa, $n \sim 0.2$, and for pressures between 70 and 300 MPa, $n \sim 1.1$. One HAN-based liquid propellant, XM46, has been investigated as a function of temperature as well. Due to the substantial increase in viscosity, cold-temperature ($\sim -50^\circ\text{C}$) burn rates were obtained without the use of any gelling agent. These cold temperature burn rates are slightly lower than the ambient temperature rates and follow a similar pressure behavior at pressures above about 30 MPa. Hot-temperature ($\sim +60^\circ\text{C}$) gelled XM46 burn rates are slightly larger than the ambient-temperature rates, again with a similar pressure behavior. Burn rates were also determined for gelled 9.1 M HAN through the 70-MPa pressure region, but no convincing evidence of a similar pressure break was observed.

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1. Introduction

The burning-rate behavior of a propellant vs. pressure is critical information for characterizing (modeling) the interior ballistics of a gun. Large-pressure exponents for burning rate can lead [1] to pressure oscillations. Moreover, pressure oscillations are of interest since they occur in most artillery systems employing a liquid propellant. Oberle and Wren [2] determined burn rates for XM46 as a function of pressure from a closed-bomb experiment. One of their desires was to obtain burn rates without gelling the liquid propellant. Their results indicated a rather large pressure exponent (2.0) for pressures from 100 to 190 MPa. Analysis of this closed-bomb data assumed that only the top face of the liquid propellant was exposed for combustion. The liquid propellant thus regressed in a planar cigarette fashion, where the surface area was assumed to remain constant.

Previously, McBratney, Bensinger, and Arford [3] noted substantial surface disturbances on liquid monopropellants for various ignition stimuli and attempted to minimize these irregularities by gelling. Egorshv, Kondrikov, and Yakovleva [4] have also used a gelling technique to study the burn-rate behavior of a variety of water-impregnated explosive compounds. The aim of the present work was to determine the pressure dependence of the burn rate of hydroxyl ammonium nitrate (HAN)-based liquid propellants by photographic observation of the regressing interface. For ambient- and hot-temperature ungelled cases, surface irregularities were seen for all pressures studied. Gelling the HAN-based liquid propellants reduced surface irregularities to a point where an essentially planar burn was established for pressures in the range from about 80 to 300 MPa. For pressures below this range, the regressing gelled surface established a pointed surface, as previously observed [5–7]. Cold-temperature burn rates for XM46 have been obtained without the need for gelling.

2. Experimental

2.1 Ambient Temperature. Windowed steel chambers capable of pressures up to about 150 and 300 MPa have been used to house liquid-propellant samples for photographic studies of their burning characteristics. An illustration of the 300-MPa maximum-pressure windowed chamber

experiment is shown in Figure 1. The internal diameter (D) of the chamber is 19 cm, and the internal volume can be varied from 1 to 12 l. The liquid-propellant samples were typically contained in rectangular acrylic cells with cross-sectional dimensions of 0.3×1.0 cm and lengths of about 4 cm. Backlighting of the sample cells was accomplished with a 300-W quartz-tungsten halogen lamp. Here, the light enters the chamber through a sapphire window of 1.27-cm clear aperture and is subsequently turned 90° by the 45° cut on an acrylic block. Photographic records of the burning behavior were recorded through a $1.27\text{-cm} \times 5.08\text{-cm}$ rectangular clear-aperture sapphire observation window by either a half-frame 16-mm camera operating at approximately 1,000 frames/second or a 200-frame/second VHS movie camera. Regression rates were determined over a 2-cm length in the middle portion of the sample cell. This 2-cm distance was denoted by scribe marks on the cell face, and these were readily observable in the photographic records. Additionally, a known length metal needle was also placed on the cell to check for possible acrylic cell dimensional changes with pressure.

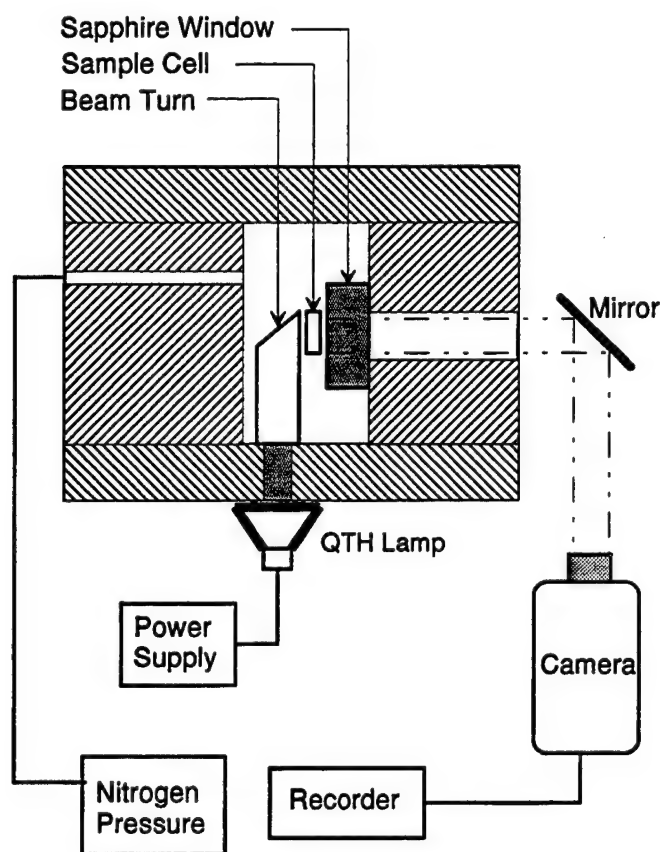


Figure 1. Sketch of High-Pressure Windowed Chamber Experiment.

2.2 Ignition. Ignition of the liquid propellant was accomplished by electrically heating a 0.1-mm-diameter nichrome wire. A number of variations of this technique were tested to see if surface disturbances could be minimized or eliminated. The bare ignition wire placed in the liquid propellant near the top surface created large surface disturbances that were present for the duration of the burn. Another variation involved coating the ignition wire with a solid mixture consisting of primarily nitrocellulose and black powder. Placement of this coated ignition wire several millimeters above the surface of the liquid propellant created a condition where the liquid propellant would be ignited by the hot combustion gases coming off the coated ignition wire. Surface effects were still seen for this case, and it also turned out to be an unreliable method of ignition. The variation that has been used for the data obtained in this report involved encasing the ignition wire with gelled XM46 and placing the same on top of the liquid XM46 contained in the sample cell. This method proved to be reliable, although varying surface disturbances were still produced and were of a sufficient magnitude to be easily observed when the ungelled liquid propellant was burned.

2.3 Gelling Agents. Several gelling agents have been tested, and those results have been discussed previously [7]. In summary, early data have been obtained by gelling with 2% Kelzan gel, and the more recent results have been obtained by gelling with 1–1.5% Rhamsam gum. Both of these gelling agents are fermentation polysaccharides and obtained from the Kelco Company.

2.4 Cold and Hot Temperature. In order to make burn-rate measurements of XM46 at cold (about -50°C) and hot (about 50°C) conditions, the present experiment needed modification. It was decided not to heat or cool the whole windowed pressure chamber because that would affect the ultimate pressure limits. Since the typical volume of propellant sample that is used per experiment is small (1.25 cm^3), hot or cold temperatures could not be maintained without the aid of heaters/coolers or a thermal mass in intimate contact with the sample. Cylindrical acrylic blocks, 4.5 cm long and 7.6 cm in diameter, were incorporated as thermal masses, and the sample cell assembly is shown in Figure 2. The internal cross section of the center of the sample cell is $0.31\text{ cm} \times 1.0\text{ cm}$. The upper part of the sample cell contains a throat with an initial cross section of $0.31\text{ cm} \times 0.30\text{ cm}$. Post-analyses of the sample cells show that this throat area has opened up to a cross section of about $0.4\text{ cm} \times 0.4\text{ cm}$. The increase in throat size is due to ablation of the acrylic

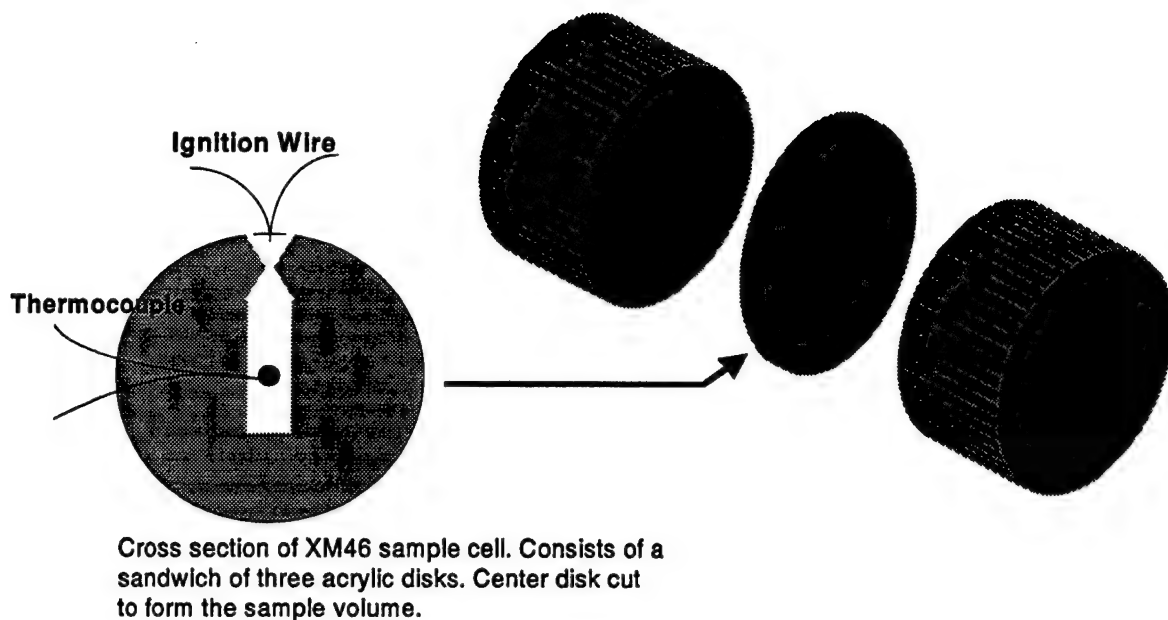


Figure 2. Sample Cell Assembly Used for Obtaining Hot and Cold Burn-Rate Measurements for XM46. The Cutout Section That Forms the Cell Is Shown Only With the Front View.

occurring during combustion. Incorporation of the throat was to minimize the perturbing ignition effects on the burn in the rectangular center portion of the sample cell. Burn rates for XM46 at the cold temperature could be obtained without gelling; thus, the increasing viscosity and/or the throat restriction provided a very beneficial effect. Incorporating a throat region in the sample cell brings up the possibility of an increased pressure within the cell. Experimental findings have led us to believe that is a negligible effect. First, since the throat area is enlarging as the burn progresses, any overpressure would diminish toward the end of the burn. However, burn rates measured at the beginning of the burn are the same as toward the end of the burn. Moreover, the sample cell design would not maintain integrity for significant overpressures.

The chromel-alumel thermocouple used to monitor the cell temperature was taped to an outside face of the sample cell in the midportion region. These three-piece sample cell assemblies are sandwiched together in a neoprene insulation sleeve and clear sealing fluids, which are composed of silicon oil or ethanol and Cabosil, and applied to the sample cell faces to provide enhanced thermal contact with the acrylic thermal masses. In addition, the ethanol/Cabosil was used to

dissolve frost buildup and thus maintain a good video observation path. Prior to an experiment, the sample cell assemblies are conditioned at hot or cold temperatures for 12 hr in a temperature-conditioning box.

The ingredients of various liquid-propellant mixtures for which data are presented are given in Table 1. The amounts are given in weight-percent. Here, the abbreviations are: TEAN (triethanol-ammonium nitrate) and DEHAN (diethylhydroxyammonium nitrate).

Table 1. Propellant Mixtures

Liquid Propellant	HAN	DEHAN	TEAN	Water
LP1845	63.2	0	20.0	16.8
XM46	60.8	0	19.2	20.0
LP4620	60.8	3.84	15.36	20.0
LP4640	60.8	7.68	11.52	20.0
LP1898	60.8	19.2	0	20.0
9.1 M HAN	60.8	0	0	39.2

3. Results

As a baseline from which to compare present data, three data sets obtained from previous work, conducted in this laboratory, are reproduced in Figure 3. These data sets are plotted on one figure since the perceived differences are minor; LGP1845 is slightly more energetic than XM46, and one data set for XM46 uses half as much of a similar gelling agent. The straight lines on the figure correspond to least-squares fits of the XM46 data for a low- and a high-pressure range. This break in pressure dependence is not as abrupt as the straight-line intersection would indicate, but the phenomena is most definite. The XM46 data sets indicate this break to be in the neighborhood of 70 MPa. Assuming an exponential burn-rate law, coefficients (A) and pressure exponents are obtained from a least-squares fit and tabulated in Table 2. Only XM46 gelled with 1% Rhamsam has been extended to 300 MPa in pressure. Here, there is some indication that another pressure break exists around 250 MPa. LGP1845 displays burn-rate behavior similar to XM46.

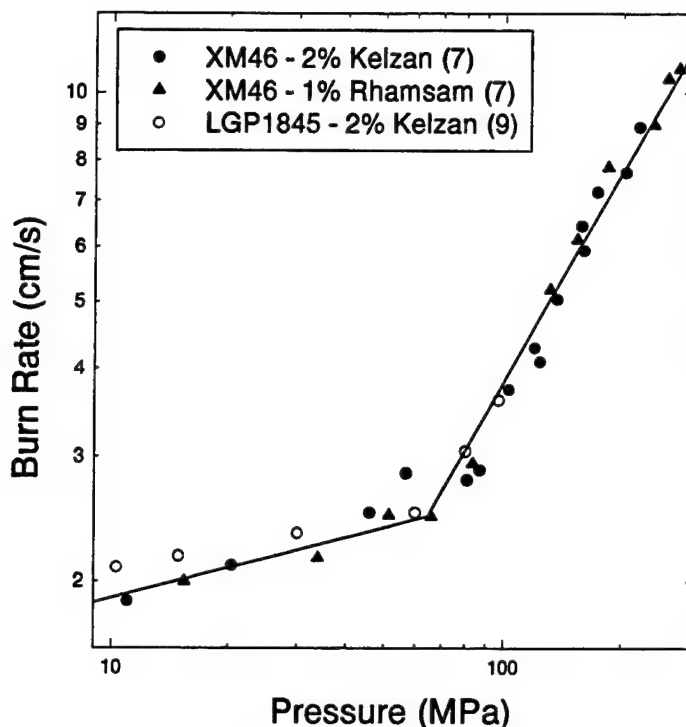


Figure 3. Windowed Strand Burner Burn-Rate Data for XM46 and LGP1845 as a Function of Pressure at Ambient-Temperature Conditions.

In order to further investigate possible mechanisms for the observed pressure break, a short time was spent in obtaining burn rates of 9.1 M HAN (a mix without the fuel component, ie TEAN fuel is replaced with water). Six experimental runs were made to determine the burn rate behavior for gelled 9.1 M HAN around the 70-MPa pressure regime. These are shown with the gelled XM46 results in Figure 4. While the burning rate for 9.1 M HAN is similar to that of the low-pressure leg for XM46, there is no apparent indication of a sharp pressure break. A least-squares fit (exponential burn-rate law) to this data results in a pressure exponent value of 0.24 (see Table 2). Here, it is seen that the presence of the fuel component has a major effect on the burn rate at pressures exceeding about 70 MPa. Further discussion of these observations is continued in the discussion section. More recently, another candidate liquid propellant (LP1898) has been formulated, with initial hopes of reducing pressure oscillations [9]. Replacement of TEAN with DEHAN in the XM46 mixture (LP1898) was thought to increase both the burn-rate and low-pressure ignitability. A comparison of gelled LP1898 and XM46 is given on Figure 5. In general, the burn-rate behavior of LP1898 is somewhat similar to XM46. There is a pressure break that appears, but at a lower pressure (about

Table 2. Fitted Values for the Burn Rates of Various Propellant Samples When Using the Exponential Form $r = Ap^n$, Where r Is in Centimeters Per Second and p Is in Megapascals. Both the Fitted Values and Uncertainty Limits Come From a Regression Analysis.

Sample	Pressure Range (MPa)	A (cm/s)	n
XM46 - 2% Kelzan	10–70	1.04 ± 0.11	0.24 ± 0.04
XM46 - 2% Kelzan	70–220	0.014 ± 0.004	1.19 ± 0.05
XM46 - 1% Rhamsam	10–70	1.27 ± 0.16	0.16 ± 0.04
XM46 - 1% Rhamsam	70–300	0.031 ± 0.008	1.04 ± 0.05
XM46 - 1% Rhamsam	70–240	0.024 ± 0.011	1.10 ± 0.09
XM46 - Ungelled - -50°C	10–70	2.29 ± 0.41	0.013 ± 0.05
XM46 - Ungelled - -50°C	100–176	0.0197 ± 0.016	1.07 ± 0.16
LGP1845 - 2% Kelzan	10–60	1.65 ± 0.01	0.10 ± 0.02
9.1 M HAN - 1% Rhamsam	30–180	1.09 ± 0.16	0.24 ± 0.03
LP1898 - 1.5% Rhamsam	10–50	1.09 ± 0.09	0.21 ± 0.025
LP1898 - 1.5% Rhamsam	70–200	0.029 ± 0.019	1.14 ± 0.13
XM46 - 1% Rhamsam ^a	20–73	1.87	0.10
XM46 - 1% Rhamsam ^a	73–190	0.017	1.2
LP4620 - 1% Rhamsam ^a	20–68	1.13	0.26
LP4620 - 1% Rhamsam ^a	68–170	0.03	1.1
LP4640 - 1% Rhamsam ^a	20–64	0.83	0.32
LP4640 - 1% Rhamsam ^a	64–170	0.032	1.24

^a Values obtained from Messina [8].

50 MPa). The burn rate of gelled LP1898, on the high-pressure leg, is about 1.5 times larger than that for XM46.

Investigations of the burning-rate behavior of XM46 at cold and hot conditions has also been undertaken [10]. It was experimentally observed that the cold (-50°C) XM46 could be made to burn with a flat surface without gelling; these burn-rate results, together with the ambient-temperature gelled data, are displayed in Figure 6. The low-pressure has considerable scatter, and the pressure exponent has large uncertainty. If the three lowest pressure points are

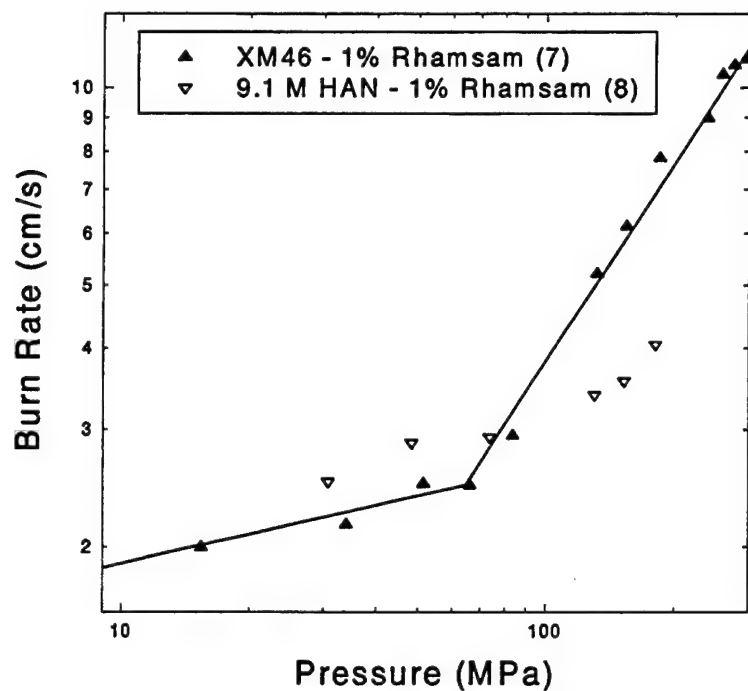


Figure 4. Burn-Rate Data Comparison Between 9.1 M HAN and XM46.

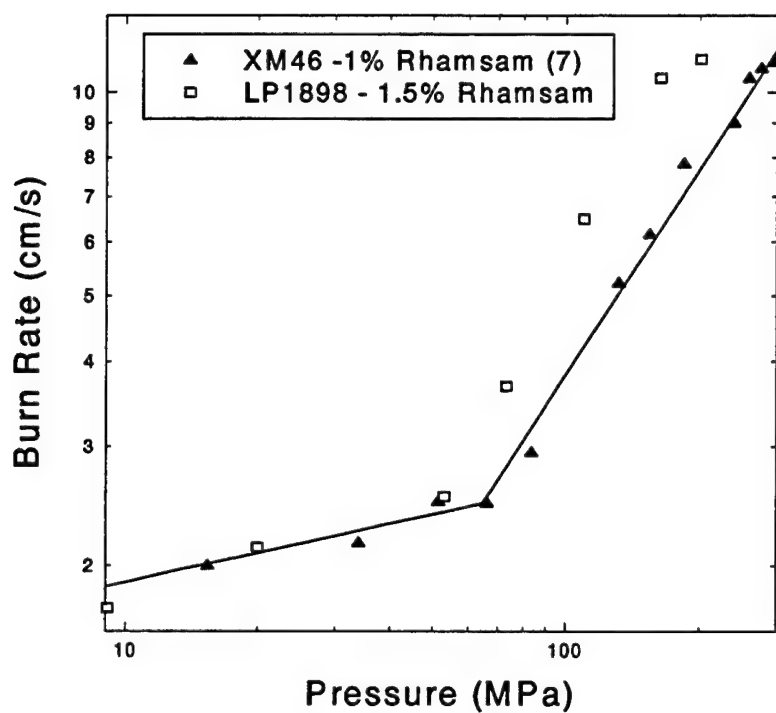


Figure 5. Burn-Rate Data Comparison Between LP1898 and XM46.

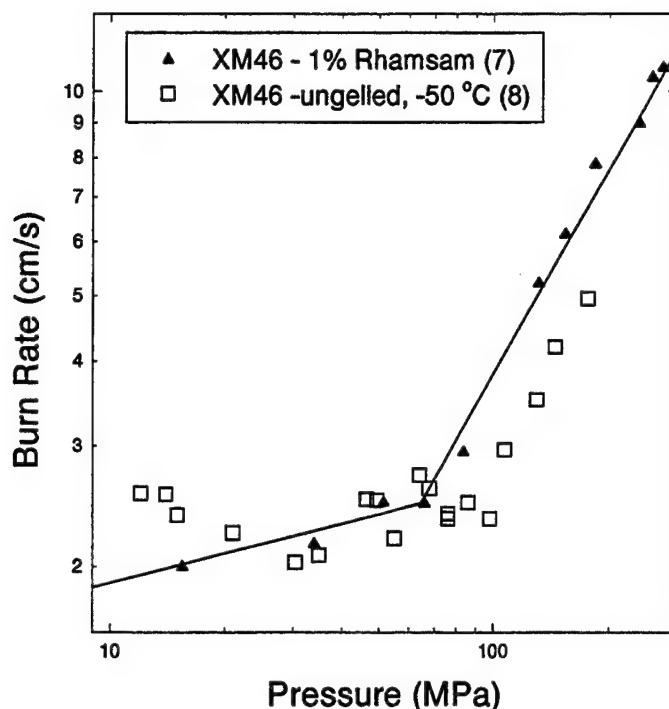


Figure 6. Comparison of the Burn Rates for Gelled XM46 at Ambient Temperature (25° C) and Ungelled XM46 at Cold Temperature (-50° C).

neglected, the ungelled data follow a trend similar to that shown for the ambient-temperature gelled case. The high-pressure-leg data has much less scatter and provides a pressure exponent (within experimental uncertainty) the same as for the ambient-temperature gelled condition (see Table 2.).

As an indication that the cold temperatures are maintained by the sample assembly for the time it takes to set up and run the experiment, an early thermocouple output for sample cell temperature is given in Figure 7. Zero time is when the sample cell assembly is removed from the temperature-conditioning box. A rapid change (decrease at 13.5 min) is observed when the windowed chamber is pressurized. This abrupt change is thought to be due to a nonuniform environment around the thermocouple connections and feedthroughs. When these feedthroughs are replaced with thermocouple material, the dip in the thermocouple trace is eliminated. As this change relaxes back, the liquid propellant sample is ignited and subsequently burns (14.3 min), giving rise to a sharp increase in temperature. After burn completion, the temperature again relaxes back to

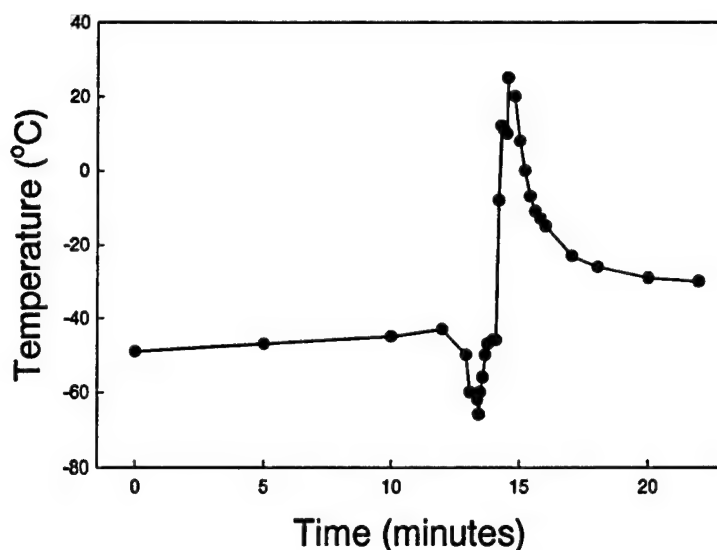


Figure 7. Temperature History of the Sample Cell Assembly for a Cold-Temperature Experiment.

values around -30°C . Other temperature histories at different pressures show similar behavior. These thermocouple data indicate that the temperature under which the experiment is carried out is typically within 5°C of the conditioning box temperature (-50°C).

Overnight conditioning of the gelled XM46 at $+65^{\circ}\text{C}$ resulted in obtaining unstable burning surfaces. However, if the thermal blocks were conditioned overnight without the sample and then the sample placed in contact with the blocks long enough to reach temperature (about 0.5 hr), then the a stable burn surface could occasionally be obtained. Apparently, conditioning the gelled XM46 at elevated temperatures for extended periods resulted in breaking down the gelling agent. Again, the temperature at which the experiment is carried out will be within 5°C of the conditioning box temperature ($+65^{\circ}\text{C}$). Only four experimental points were obtained for this study, and, thus, pressure breaks are not obvious. If one uses the ambient temperature burn-rate behavior as a guide, then there are indications that the hot burn rates (Figure 8) are close to the ambient values on the low-pressure leg; the pressure break appears to occur at somewhat lower pressures, and the values for the high-pressure leg are larger than the ambient case.

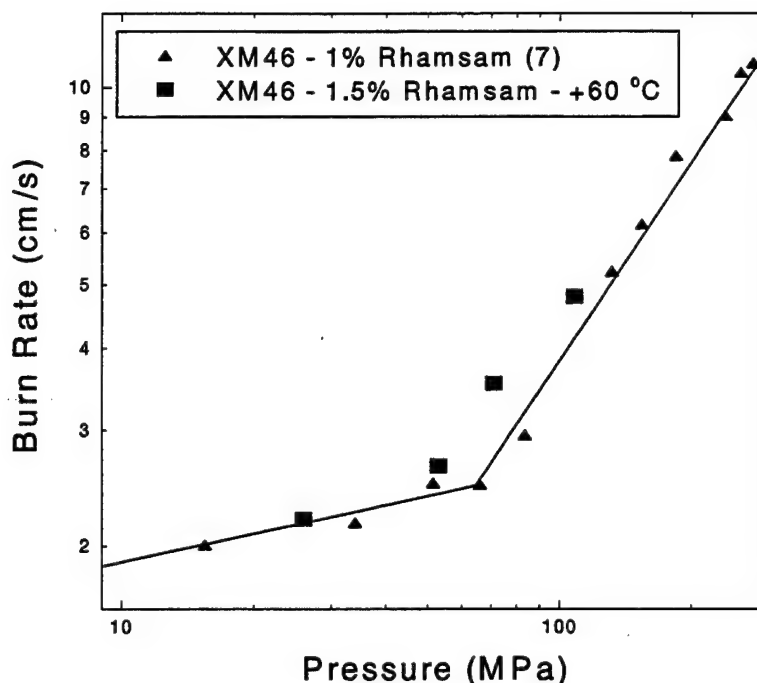


Figure 8. Comparison of the Burn-Rate Behavior Between Gelled XM46 at Ambient and +60° C.

4. Discussion

Consistent trends for ambient- and hot-temperature liquid-propellant burn rates have only been obtained by gelling. No other investigators have measured burn rates by visualization of the burning surface over this pressure range. As mentioned previously, Oberle and Wren [2] measured burn rates of ungelled liquid propellant with a closed-bomb technique. Here, the geometry of the burning surface has been assumed planar in order to extract burn rates from pressurization rates. Results of McBratney and Vanderhoff [7] are in disagreement with these extracted burn rates. Recently, Messina [8] has measured the burn rates for gelled XM46, LP4620, and LP4640 using a pressure-fluctuation technique [11]. Results of these measurements are reported in Table 2. One of the objectives in their study [8] was to check against the work of McBratney and Vanderhoff [7, 10]. Reasonable agreement was found for the burn rate of gelled XM46 over the pressure range for the comparison. The values obtained by Messina were somewhat larger than those of McBratney and Vanderhoff. The break in the pressure exponent occurred at similar pressures. A direct comparison cannot be drawn for LP1898 since Messina measured mixtures of XM46 and LP1898

and we measured pure LP1898. However, trends can be pointed out. LP1898 does not have a substantially higher burn rate than XM46. Through the pressure range studied, LP1898 burns, at most, 1.5 times faster than XM46. The burn rates for the mixtures studied by Messina fall within these bounds.

Over the course of these studies, there has been concern about gelling the liquid-propellant samples and the effect this may have on the burning rate. Evidence that gelling has a minimal effect on the burning rate comes from windowed chamber studies of XM46 at cold (-50°C) conditions, where burn-rate measurements were obtained without the need for gelling. These cold-temperature ungelled burn-rate results are about what one would calculate from taking ambient-temperature gelled values and applying a moderate-temperature-sensitivity correction. That is, the temperature sensitivity (σ_p), defined as $\{\partial \ln r / \partial T\}_p$, calculated from the burn-rate results shown in Figure 6 is $0.005/^{\circ}\text{C}$ for the high-pressure leg. According to Kubota [12], this value lies in the middle of the range of temperature-sensitivity values for conventional solid propellants. Actual temperature sensitivity values were not computed for the low-pressure (low-pressure exponent) leg due to large data scatter. However, one could speculate that the ambient-temperature and cold-temperature data were about the same—hence, no temperature sensitivity. This type of behavior has been observed. Experiments on binary mixtures of magnesium and sodium nitrate (candidate propellant for air augmented rockets) show that, for mixtures where the pressure exponent of the burn rate is small, the temperature sensitivity is also small [13]. Conversely, in the same study they show larger temperature sensitivities for mixtures where the pressure exponent of the burning rate is larger. While a case has been made for gelling having a minimal effect on the burn rate, the obvious set of measurements have not been made. Measurements for gelled XM46 at -50°C should be made and compared with the -50°C ungelled XM46 work.

The reason that liquid XM46 burned with a reasonably flat surface at the cold temperatures (nominally -50°C) was assumed to be due to increases in viscosity. Over the temperature range from $+25^{\circ}\text{C}$ to -55°C , the dynamic viscosity increases from about 0.1 to 35 P. These viscosity data were obtained by Bair [14] for a pressure of 69 MPa. Zeldovich et al. [15] discuss combustion instabilities in liquids as related to viscosity and state:

“When the viscosity of the burning liquid is sufficiently high (on the order of 1 P) the stabilizing factor will be the viscosity, rather than the surface tension.”

Moreover, a stability criteria against hydrodynamic perturbations is given [15] for burning liquids under the influence of gravity. This relationship is

$$m^3 < gv(3\rho_1\rho_2)^{3/2} \text{ and } m = \rho_1 u_n$$

for the case where the burning surface regresses downward. The symbols, units, and estimated values for liquid XM46 are given in Table 3. Using these values, the stability criterion is met for kinematic viscosity values greater than about 0.18 cm²/s or dynamic viscosity values of about 0.26 P. For XM46, at atmospheric pressure and above, the stability criterion is attained when XM46 is cooled to temperatures below 0° C.

Table 3. Some Thermophysical Quantities for XM46 at +25° C and 70 MPa

Quantity	Symbol	Units	Values for XM46
Mass flux of propellant	m	g/cm ² s	3.6
Density of liquid propellant	ρ_1	g/cm ³	1.45
Denisty of combustion products	ρ_2	g/cm ³	0.0945 ^a
Acceleration of gravity	g	cm/s ²	980
Dynamic viscosity	ν_d	P	0.09
Kinematic viscosity	ν	cm ² /s	0.06
Burning velocity	u_n	cm/s	2.5

^a Calculated from thermochemical equilibrium code.

Hot-temperature burn rates (about +60° C) for XM46 could only be obtained by gelling. Initially, experiments were conducted at +60° C using 1% Rhamsam gum as the gelling agent. Videos of the burn showed a surface destabilized behavior consisting of a negative burn cone with wavelets progressing up the sides of the cone. The gelling concentration was increased from 1.0 to 1.5%. This change, along with minimizing the time the gelled sample was kept at the hot

temperature, provided stable burning behavior. The burn rates obtained were slightly larger than the gelled ambient results, with a similar pressure behavior.

The pressure break provides a convenient separation point for discussing possible differences in the combustion mechanisms of gelled XM46. Although the pressure break is depicted as a sharp change with the lines drawn in the figures, the actual transition from one pressure dependence to another is probably smoother, occurring over a finite-pressure regime. For pressures above about 70 MPa, the gelled regressing surface appears planar, with a sharp line defining the burning surface. At pressures below about 70 MPa, two distinct patterns emerge. In the pressure range from about 25 to 70 MPa, the planar surface develops a slightly convex nature as the pressure is decreased, but the gas phase region above the surface remains transparent. Moreover, the line defining the burning surface is less distinct. Below about 25 MPa, the initially planar regressing surface develops inclined surfaces and a dark residue remains. Here, a wedge-shaped surface is observed where the gas phase area above is rendered mostly opaque due to residue coating the sample cell walls. This phenomena has been previously observed by McBratney [5] and is consistent with one of the major conclusions of Vosen [16], who deduced that the combustion of XM46 is a two-step process. The first step is liquid-phase decomposition of HAN, and the subsequent step is the decomposition of TEAN in the gas-phase HAN products. Vosen's conclusion was predicated on a post-analysis, where it was found the that TEAN was the primary burn-residue ingredient.

In their studies of LP1845 (liquid propellant almost identical to XM46) Zhu and Law [17] report that the propellant explosion is initiated by a liquid-phase reaction of the HAN component. Thus, evidence is given for a condensed-phase HAN reaction as being the controlling or rate-limiting reaction for XM46 at low pressure (0.1 MPa). Additional evidence supporting this mechanism is the observed weak-pressure dependence of the burn rate for both gelled XM46 and ungelled XM46 (-50°C) at pressures below about 70 MPa. At pressures above 70 MPa, the pressure exponent attains values close to 1, which is suggestive of a controlling bimolecular reaction.

A few burn-rate measurements on gelled 9.1 M HAN were performed to further characterize the pressure break region. These measurements suggest no pressure break, a major change in behavior

due to the absence of the fuel component, TEAN, or DEHAN. Reactions controlling the burn rate remain weakly pressure-dependent throughout the pressure range investigated (30–200 MPa). Kounalakis and Faeth [18] have theoretically studied critical combustion properties of LGP1845 and XM46 and estimated a critical combustion pressure of 250 ± 125 MPa. This value is larger than the observed pressure break in the burn rate. These findings, together with the absence of a pressure break region for 9.1 M HAN, support the position that the pressure break is not a manifestation of critical point behavior.

5. Summary

In this report, burn-rate data have been presented for six different aqueous HAN-based mixtures. These data have been obtained as a function of pressure and, in one case, temperature as well. The pressure range covered extends from low pressure into gun condition pressures (10–300 MPa). Two distinctly different pressure regimes were found. At pressures below about 70 MPa, the burn rate is weakly pressure-dependant, whereas, at pressures from about 70–250 MPa, the burn rate is approximately linearly dependent on the pressure.

If these materials were to be seriously considered for propellant use at some future time, the following suggested study is proposed. While the burn-rate behavior of ungelled XM46 at -50°C is very similar to the behavior of gelled XM46 at ambient temperature, a more conclusive test would be to measure the burn rates of XM46 gelled and ungelled at -50°C .

If the results of this comparison showed identical behavior, then closed-bomb techniques for the burn rate measurement could possibly be developed. Gelled liquid propellant might be shaped into a disk and uniformly ignited in a closed-bomb vessel. Run-to-run reproducibility, as well as agreement with the optical strand burner data, would be necessary before applying the closed bomb to other samples or over different pressure ranges. The benefits of having a validated procedure for closed-bomb testing would be that the pressure dependence of the burn rate could be obtained in one run. Moreover, the pressure range could be extended further into the gun-pressure regime, 600 MPa.

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